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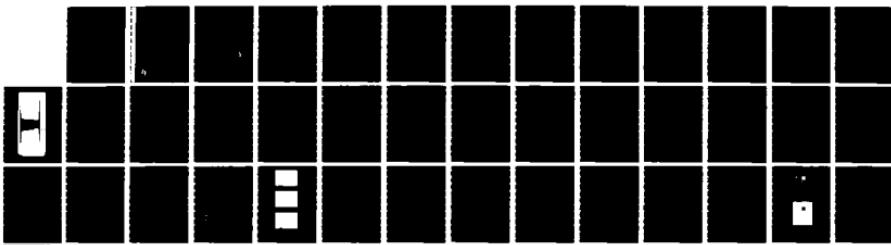
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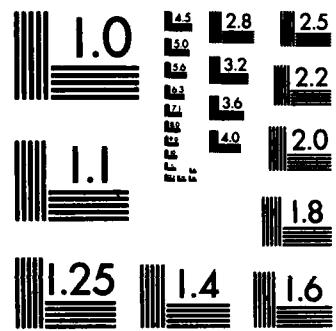
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EFFECTS OF ENVIRONMENT OR STORAGE CONDITIONS ON RADIOGRAPHIC FILM

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by

Russell D. Williams

FINAL REPORT

SwRI Project 15-7325-001
Contract No. F41608-82-C-2101 (A002)

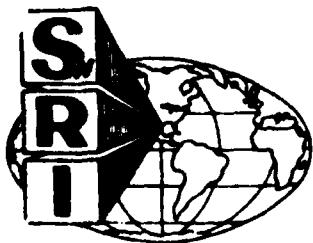
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December 1983

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John R. Barton

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Instrumentation Research Division

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ABSTRACT

This report covers the approach, procedures and technique employed in evaluating sensitivity and base plus fog in old and new radiographic films stored in simulated tropical, desert, arctic and ambient environments. A simple field test for film qualification is described and a design for the step wedge used in the test is included. Also included is a description of a laboratory test for film noise using electronic scanning of an optical image to determine frequency content of this image. Recommendations are made for extension of film life through testing, as well as recommended storage and shipping conditions for life extension.

I. INTRODUCTION

A number of factors influence the results obtained using radiography for critical aircraft component inspection procedures. One of the more important factors is the quality of the x-ray film. This is particularly true in applications where high resolution and high sensitivity are essential, for example in those inspections for detecting fine cracks. Quality of the film degrades with age and the degree of deterioration is strongly influenced by the storage environment. For example, two industrial x-ray films stored for identical periods but each under different environmental storage conditions may produce completely different results when applied to the same inspection problem. Results may range from completely satisfactory to useless. In some cases aged and/or deteriorated film may not be suitable for fine crack detection but may yield entirely satisfactory results on less critical applications. Despite the foregoing, radiographers have no knowledge of the storage history of films they receive from warehouse stock other than the manufacturer's expiration date or life extension date as approved after tests at depot level.

Accordingly, this investigation was undertaken: (a) to determine the effect of aging under various environmental storage conditions on the usefulness of industrial x-ray film; and (b) to develop a simple, field applicable, test for qualifying x-ray films for the various inspection requirements encountered.

The results of accelerated environmental storage testing performed on this program demonstrate that film well within the manufacturer's expiration date can be deteriorated by adverse environmental storage conditions to the point that the integrity of the inspection procedure is compromised.

This report details the simulation of tropical, desert, arctic and laboratory storage environments typical of those which realistically may be experienced at Air Force bases throughout the world and presents a detailed analysis of the various films stored in these simulated environments. The report also lists results of tests performed on both old and new films received from several Air Force NDI field laboratories as well as old, out of date, films stored under known conditions at SwRI.

The report also includes recommendations on storage and transportation requirements and gives the mathematical and graphic procedures for performing a simple field test for film sensitivity involving use of a step wedge. Two additional approaches for determination of film quality are described and discussed.

II. LITERATURE SEARCH

A comprehensive literature search was performed to determine if any studies of this type had been previously accomplished. In addition to using the facilities and data base of NTIAC, at SwRI, other data bases searched were:

Engineering Index
Chemical Abstracts
Science Abstracts (INSPEC)
NTIS.

Relatively few "finds" of interest were made, primarily in the area of photographic films. Since the chemistry of photographic, and radiographic films is similar, copies of articles with subject titles of interest were ordered and reviewed.

The most meaningful articles received covered photographic film characteristic changes induced by low and high humidity and for elevated temperature.

None of the articles reviewed were of a general nature and were directed more to special purpose films for other than radiography. Since a parallel review of manufacturers literature failed to discover any previous similar research, the decision was made to continue with all phases of the planned study.

A bibliography of the articles requested and received follows. Copies of these articles will be included with other project residuals and will be delivered upon instructions received at the end of the contract.

Bibliography:

1. Low Humidity Induced Fog in Photographic Films, Kathren, R.L. and Samardzich, B.G., Hazards Control Progress Report No.25, UCRL.
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7. Effects of Moisture on Photographic Sensitivity, Babcock, Michrina, McCue and James, Photographic Science and Engineering, Vol.17, No.4, Jul-Aug, 1973.
8. Kinetic Analysis of the Structuralization of Hardened Gelatin Layers by Studying Swelling Phenomena, Claes, Boulonne and Beels, Photographic Science and Engineering, Vo.22, No.1, Jan-Feb, 1978.
9. Humidity and Photographic Sensitivity, Tull, A.G., Symposium on Photographic Sensitivity, Cambridge, U.K., Sept. 1972.
10. Estimation of Images Degraded by Film-Grain Noise, Naderi and Sawchuk, Applied Optics, Vol.17, No.8, 15 Apr 1978.

III. FILM SENSITIVITY

Film contrast is defined as the slope of the characteristic curve (i.e. $\Delta D/\Delta \log E$) in the area of a particular desired density. These published characteristic curves are generally applicable only to fresh films not exposed to questionable storage environments.

For rapid determination of new and aged film sensitivity, a short method using the slope of a density vs. thickness ($\Delta D/\Delta t$) curve was employed throughout this investigation. This method is valid since it is also utilized as a part of the basic procedure for deriving the characteristic curves for X-ray films.

To establish a uniform basis for accept/reject criteria, the following were adopted as minimum requirements throughout the evaluations formed during this investigation.

1. A minimum sensitivity of 2% at an average density of 2 H&D units is acceptable for critical inspections.
2. The average minimum density difference discernable by trained radiographers is 0.04 H&D unit.

If we now expose an X-ray film under an object (specimen) 0.500 inch thick with a 2% thickness defect, the slope of a density versus thickness curve to image this defect should be;

$$\frac{\Delta D}{\Delta t} = \frac{0.04}{0.02 \times 0.500} = 4.0$$

By applying the above formula we may then define the slope for a family of curves based on a percentage specimen thickness.

<u>Sensitivity %</u>	<u>Slope $\Delta D/\Delta t$</u>
1	8.0
2	4.0
3	2.7
5	1.6

Figure 4 illustrates a family of curves which may be used to determine film sensitivity based on the above criteria. A useful overlay may be made from Figure 4 by making a Xerox transparency which may then be compared directly to a plotted curve of density vs. thickness as described below.

Figure 1 is a double ended laboratory step wedge, designed for work in developing X-ray film characteristic curves. Due to the greater inherent accuracy of this step wedge as compared to a single ended type it was used throughout this program in order to resolve the anticipated very small changes in characteristics. As may be seen, this step wedge contains a total of 32 steps in 0.0625 inch increments. Figure 2 is a photographic reproduction of an actual radiograph of this step wedge and is approximately 60% of original size. It is shown here for illustrative purposes only. In obtaining data,

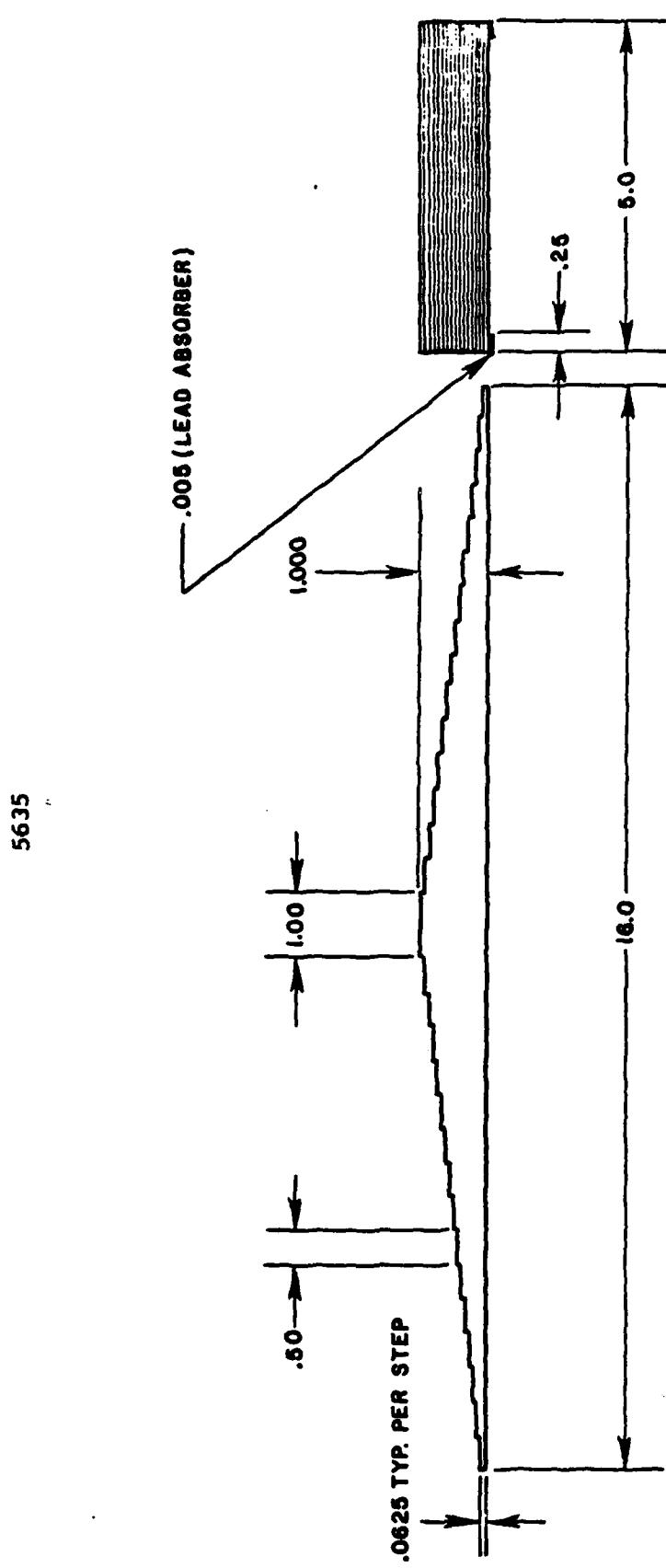


FIGURE 1. Aluminum Step Wedge

2024 Aluminum Alloy

6705

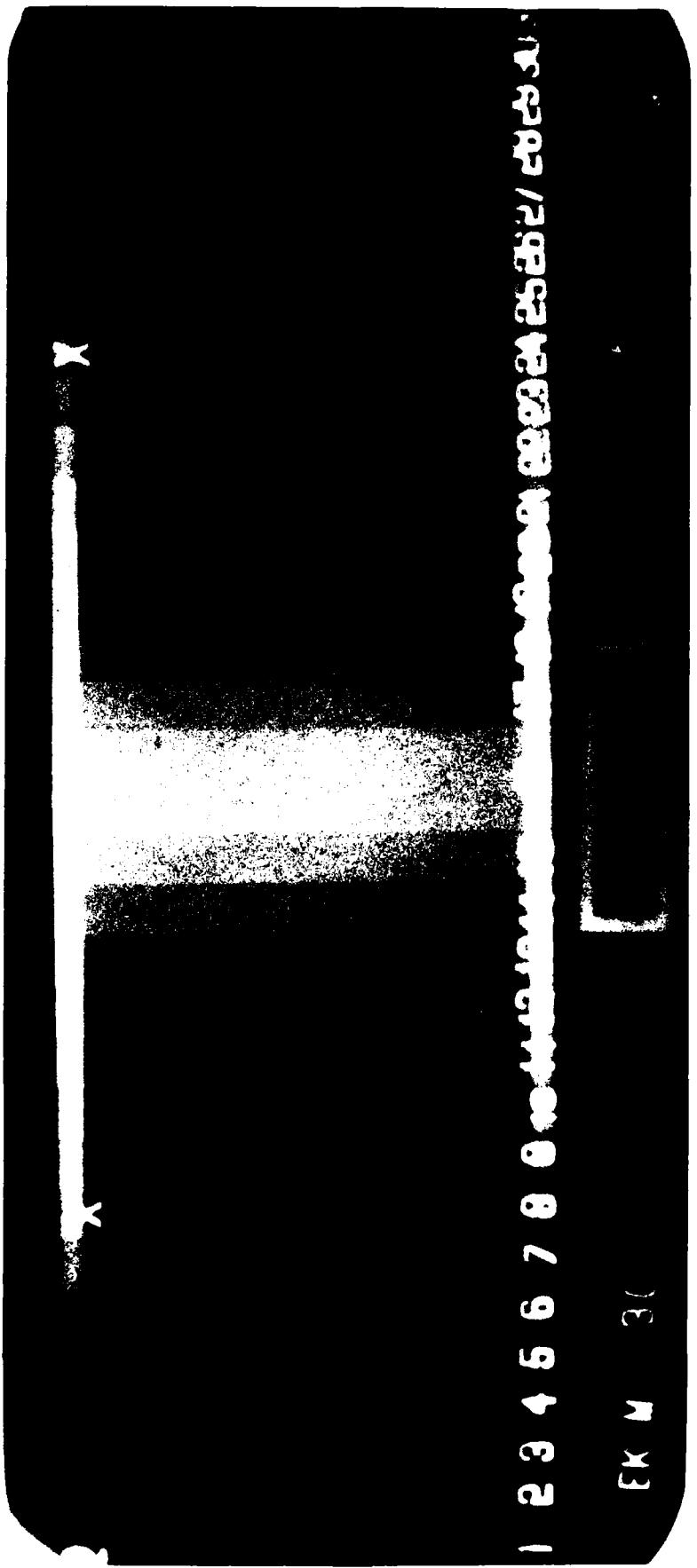


FIGURE 2. TYPICAL RADIGRAPH OF LABORATORY STEP WEDGE

four density measurements were made for each of the 32 steps. These density readings were then averaged, first within the individual steps and then end to end for equal step thickness. This was done to minimize exposure differences caused by inherent X-ray tube geometry. The resulting densities were plotted vs. step thickness on K&E #46-1323, 10 x 10 to 1/2 inch, graph paper as shown in Figure 3. After constructing a best fit curve, connecting all plotted points, the overlay described below may be used to directly determine approximate sensitivity of the film. An alternate, and more accurate, method is to choose two points on the curve equally spaced about a desired density, determine the thickness of material at each of these densities and calculate sensitivity directly.

A useful formula to determine the actual percentage sensitivity achieved is:

$$\% \text{ Sensitivity} = \frac{(\text{Average minimum observable } D)}{(\text{Median thickness}) \times (\text{slope})} \times 100 \quad (1)$$

where: Average minimum $D = 0.04$

$$\text{Median thickness } t_m = \frac{t_C + t_B}{2}$$

$$\text{Slope} = \Delta D / \Delta t = - \frac{D_B - D_C}{t_B - t_C}$$

where the minus sign is utilized to insure that the resultant slope of the tangent to the density curve is a positive number.

For example in Figure 2 we have

$$\Delta D / \Delta t = \frac{2.2 - 1.8}{.615 - .500} = \frac{0.4}{.115} = 3.47$$

and

$$t_m = \frac{.615 + .500}{2} = .558$$

then

$$\begin{aligned} \% \text{ Sensitivity} &= \frac{0.04}{.558 \times 3.47} \times 100 \\ &= 2.0658\% \\ &= 2.1\% \text{ at a density of 2.0 (Point A)} \end{aligned}$$

In the example above, although 16 density steps were available, only 6 density points were plotted in the range of interest (i.e. ~ 1.0 to 3.5 H&D units) as these are all that is required to produce an accurate curve. In the event that any density point would fall outside of this curve, the other density readings could be utilized for corrections.

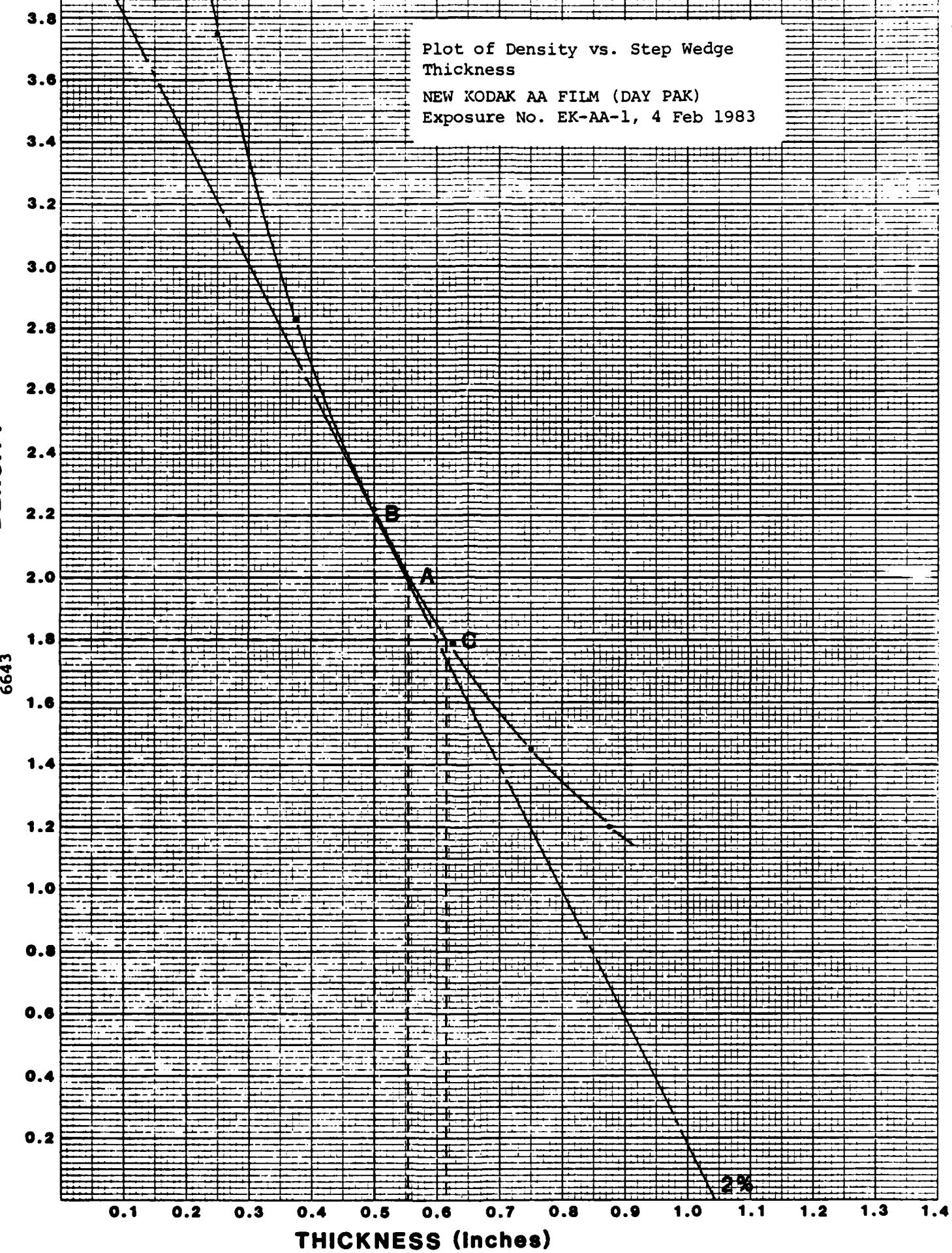


FIGURE 3. GRAPH OF DENSITY VS. THICKNESS

Referring again to Figure 5, in the column headed % Sensitivity are sensitivities calculated for each of the data pairs to their right. These percentages were calculated directly from the data using the formula (1.) above. This is a more accurate method since it eliminates the intermediate steps of plotting and extracting data from the curve. It also serves to illustrate the sensitivity increase with increasing density.

SENSITIVITY OVERLAY

Use with Plotting
sheet K & E #46-1323
10 X 10 to 1/2 inch.
Subdivided as per
Figure 2.

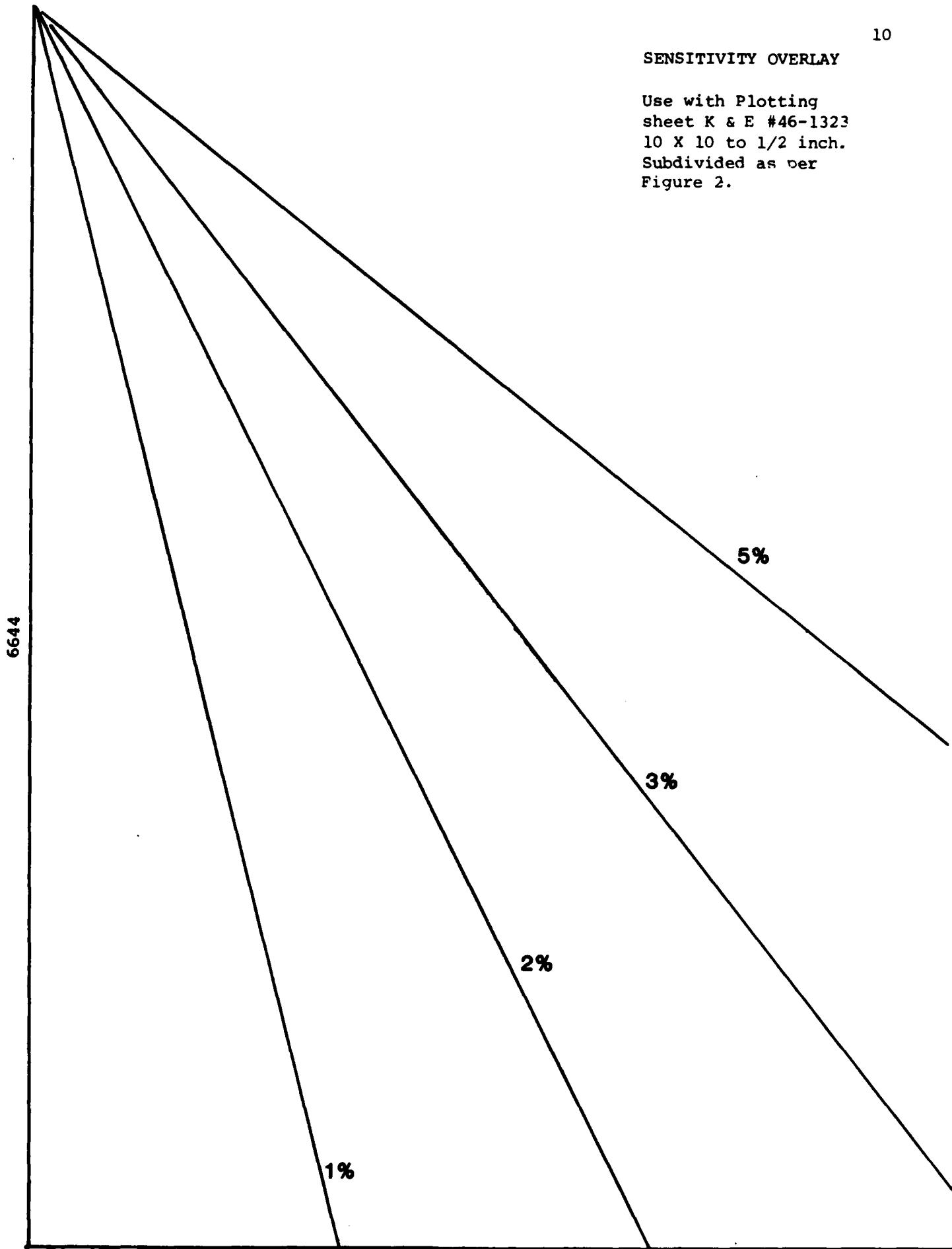


FIGURE 4. SENSITIVITY OVERLAY

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EK-AA-1

Day Pak New

Step	#	t _s	Day Pak New				Avg.	%	Avg.	%
			1	2	3	4				
1	5.32	5.33	5.31	5.32	1		5.32		5.32	.
2	5.03	5.02	5.02	5.02	2		5.02		5.04	
3	4.36	4.34	4.35	4.34	3		4.35		4.38	
4 .250"	3.72	3.74	3.73	3.72	4		3.73	1.73	3.75	
5	3.22	3.23	3.23	3.23	5		3.23	1.76	3.24	
6 .375"	2.81	2.82	2.83	2.82	6		2.82	1.85	2.83	
7	2.48	2.50	2.50	2.49	7		2.49	1.89	2.50	
8 .500"	2.21	2.22	2.23	2.22	8		2.22	2.03	2.22	
9	1.98	1.99	1.99	1.97	9		1.98	2.09	1.99	2.1%
10 .625"	1.78	1.78	1.77	1.77	10		1.78	2.10	1.79	
11	1.60	1.60	1.60	1.60	11		1.60	2.16	1.61	
12 .750"	1.44	1.44	1.45	1.45	12		1.45		1.45	
13	1.31	1.31	1.31	1.30	13		1.31		1.32	
14 .875"	1.19	1.19	1.20	1.19	14		1.19		1.20	
15	1.08	1.09	1.09	1.08	15		1.09		1.09	
16	.99	1.00	1.00	1.00	16		1.00		1.00	
16	.99	1.00	1.00	1.00	16		1.00		1.00	
17	1.09	1.10	1.09	1.09	15		1.09			
18	1.20	1.20	1.19	1.19	14		1.20			
19	1.31	1.32	1.32	1.32	13		1.32			
20	1.44	1.46	1.46	1.45	12		1.45			
21	1.61	1.61	1.61	1.60	11		1.61			
22	1.79	1.79	1.80	1.78	10		1.79			
23	1.99	1.99	1.99	1.99	9		1.99			
24	2.22	2.22	2.22	2.22	8		2.22			
25	2.50	2.51	2.50	2.49	7		2.50			
26	2.86	2.85	2.83	2.82	6		2.84			
27	3.28	3.25	3.24	3.22	5		3.25			
28	3.79	3.78	3.76	3.72	4		3.76			
29	4.44	4.43	4.36	4.35	3		4.40			
30	5.08	5.06	5.04	5.02	2		5.05			
31	5.31	5.30	5.30	5.31	1		5.31			
		1	2	3	4	Fog Avg.				
FOG		.16	.16	.16	.16	.16				

FIGURE 5. TYPICAL DATA SHEET

IV. ENVIRONMENTAL TESTING

A. Simulation of Environments

The environments chosen for simulation were selected to cover the wide range of storage and usage conditions likely to be encountered at Air Force field stations throughout the world.

These storage conditions were;

1. Tropical (95° - 105°F, 95% to 100% R.H.)
2. Desert (115° - 125°F, 5% to 20% R.H.)
3. Arctic (0° to -30°F, Ambient Humidity)
4. Ambient (Standard Laboratory, 76° - 82°F at approximately 50% R.H.)

In addition to the above a control group of film was placed in refrigerated storage (34° to 44°F, Ambient Humidity) as backup in the event any unexplained difficulties were encountered during the testing phase of this program.

Storage facilities for the simulated Tropical and Desert conditions were identical in construction, both being essentially plywood cabinets containing ventilated racks for film storage, a recirculating air system and a heating and air treatment chamber. Both were thermostatically controlled to maintain the desired temperature. High humidity (>95% R.H.) was obtained in the tropical storage container by placing a large surface area water pan over the heating unit. In the desert storage container dessicant trays, with dessicant changed weekly, were used to maintain an average of 12% R.H.

The Arctic storage facility was a standard refrigerator/freezer, leased for the project duration, and capable of maintaining a freezer temperature of -20°F. The refrigerator section was utilized for storage of the control group of films.

All films used in the environmental testing program were new, fresh and from three different manufacturers; Eastman Kodak, Dupont and Gevaert. Other films tested during this program were from various Air Force units throughout the world and out of date films, remaining from a previous research program, which had been stored under known conditions.

B. Exposure Technique and Processing

X-ray exposures were made with a ANDREX 150KVP, 5MA, self rectified unit. Exposure timing was accomplished using the internal X-ray unit timer. A fixed 48 inch source to film distance was used throughout the testing, as well as a fixed X-ray energy of 110 kilovolts. A laboratory step wedge, (see Figure 1) was used for all exposures and exposures were made against a lead backing surface to minimize scattering. Exposures were carried out in a lead lined steel enclosure with safety interlocks, qualified under Texas State regulations as an exempt radiation facility.

All X-ray exposures were hand processed in standard deep tanks using stainless steel film hangers. Film development was carried out at 68°F, 6 minutes using Kodak chemistry.

After initial testing of the new films, the films were divided and placed in the various storage environments. Subsequently two films of each type, packaging and manufacturer were removed from storage at scheduled intervals and were allowed to stabilize at room temperature 24 hours before exposure and processing. One of these films was not exposed but processed along with the exposed film to determine base plus fog level changes.

New films studied during this program and their respective expiration dates were:

<u>Mfgr.</u>	<u>Film Type</u>	<u>Packaging</u>	<u>Expiration Date</u>
Kodak	Type M	Bulk	March 1984
Kodak	Type M	Redipak	Jan 1984
Kodak	Type AA	Redipak	Jan 1984
Kodak	Type AA	bulk	Feb 1984
Dupont	NDE 55	Bulk	June 1985
Dupont	NDE 55	Day Pack	Apr 1985
Dupont	NDE 75	Day Pack	Nov 1984
Dupont	NDE 75	Bulk	Sept 1984
Gevaert	D4	Bulk	July 1984
Gevaert	D7	Bulk	July 1985

C. Test Results

Initial film exposures on both old and new film were started on 3 February 1983 and 4 February 1983, respectively. All new film was placed in the simulated storage environments on 28 February 1983. Withdrawal, exposure and processing of these films was accomplished on the following dates.

14-15 March 1983
 28-29 March 1983
 26-27 April 1983
 27-28 June 1983

Results of the initial testing of new film are tabulated below.

Film Type	Packaging	Base + Fog	Sensitivity (Percent)	
			D \geq 2.0	D \geq 2.5
Kodak Type M	Day Pack	0.14	1.8	1.4
Kodak Type M	Bulk, Interleaved	0.14	1.7	1.5
Kodak Type AA	Day Pack	0.16	2.1	1.9
Kodak Type AA	Bulk, Interleaved	0.16	2.3	2.0
Dupont NDT 55	Day Pack	0.15	1.9	1.7
Dupont NDT 55	Bulk, Interleaved	0.15	2.0	1.8
Dupont NDT 75	Day Pack	0.27	2.7	2.5
Dupont NDT 75	Bulk, Interleaved	0.22	2.1	2.0
Gevaert D4	Bulk, Not Interleaved	0.14	2.2	2.0
Gevaert D4	Bulk, Not Interleaved	0.14	2.0	1.8

Base plus fog densities shown above are an average of four density readings from an unexposed film processed simultaneously with the film exposed under the step wedge. Percent sensitivities were calculated using density data directly, without subtracting base plus fog, since this constant difference would not affect the slope of the density versus thickness curve. A sample data page as used in this study is shown in Figure 5.

An example of how the sensitivities were derived is shown below.

$$\% \text{ Sensitivity} = \frac{(\text{Minimum discernable density})}{(\text{Median step thickness} \times \text{slope})} \times 100$$

Assuming a minimum discernable density of .04 H&D units and using the data of Figure 3 we obtain the following;

For a density of ~ 2.0 , we obtain from the data $D = 1.99$ at step #9, which is $9/16$ inch thick $= 0.562"$ = median thickness.

For determining slope ($\Delta D/\Delta t$) we obtain data from step 8 (0.500"), $D = 2.22$ and step 10 (0.625"), $D = 1.79$ and

$$\text{The slope is } \frac{\Delta D}{\Delta t} = \frac{2.22 - 1.79}{0.625 - 0.500} = \frac{0.43}{.125} = 3.44$$

then

$$\frac{0.04}{0.562 \times 3.44} \times 100 = 2.069 \approx 2.1 \text{ @ } D \approx 2.0$$

All film sensitivities throughout this study were calculated by the method described above. To insure accuracy in the listed results, calculations for sensitivity were made for as many data pairs, both above and below a density of 2.0, as considered necessary as a check on data consistency. It is more accurate than plotting in that data are used directly, otherwise the multiple steps of plotting the data, drawing a best fit curve and then extracting quantities from this curve may lead to errors.

1. Tropical Conditions

This proved to be the most detrimental to radiographic films, however, short term exposure, less than two weeks, may be acceptable since no measurable changes in base plus fog or sensitivity were measurable. All films in this storage environment eventually failed during the four month testing period. The following is a chronological listing of film response

Period 1-15 March 1983

No measurable changes on all films

Period 15-29 March 1983

Gevaert D-7 Slight sticking of emulsions, however,
No apparent change in sensitivity slight increase in fog level

Gevaert #4 Emulsions totally stuck unable to test.

Dupont NDE 55 Slight water spotting
base plus fog = 0.16 Sensitivity 2.1 - 2.3%

Dupont NDE 75 Gross water spotting
Sensitivity 2.6%; Base plus fog to 0.27

Kodak M. base plus fog slight increase
Sensitivity 2.0% @ D ≈ 2.0, Minor water spotting

Kodak AA base plus fog slight increase
Sensitivity 2.3 - 2.4% @ D ≈ 2.0, Minor water spotting

Period 29 March - 27 April 83

Gevaert D4 & D7 Unable to test due to sticking.

Dupont NDE 55 Severe water spotting
Bulk Sensitivity 2.8% @ D ≈ 2.0
Day Pack Sensitivity 2.4% @ D ≈ 2.0

Dupont NDE 75 Severe water spotting
Bulk Sensitivity 2.6% @ D ≈ 2.0
Day Pack Sensitivity 2.8% @ D ≈ 2.0

Kodak M Gross water spotting on bulk, minor water spotting on Day Pack
Bulk Sensitivity 2.1% @ D \geq 2.0
Day Pack Sensitivity 2.1% @ D \geq 2.0

Period 27 April 83 - 28 June 83

Dupont NDE 55 Severe mottling and base plus fog to 3.9, both packagings.

Dupont NDE 75 Severe mottling, unable to extract data.

Kodak Both M and AA showed severe mottling, film sticking to paper, unable to reduce data.

In addition to the above noted changes, it was also discovered that all film suffered a loss of speed as indicated by progressively lower densities within a designated step over the period of testing.

2. Desert Conditions

No measurable changes in sensitivity were observed for any of the film in this storage environment. Slight increases in base plus fog densities were noted in the following film;

Gevaert D4 0.14 increased to 0.17

Gevaert D7 0.14 increased to 0.18

Kodak M 0.14 increased to 0.25 - 0.30

Kodak AA 0.16 increased to 0.34 - 0.40

It was discovered after testing on 27 April 1983 that the base plus fog films for Dupont 55 and 75 films had inadvertently been light struck. Investigation revealed that all remaining Dupont films had been affected. Since only one subsequent test had been scheduled, testing was discontinued for these Dupont films.

3. Arctic Conditions

Within the limits of accuracy of these experiments no changes in sensitivity or base plus fog density were noted.

4. Ambient (Laboratory) Conditions

No changes in sensitivity or base plus fog levels in any of the new films was observed.

In addition to the new films used in this investigation, old films were on hand from a previous project*. These films all had passed the manufacturer's expiration date, and all had been stored under average laboratory conditions since the completion of the original work. Storage conditions for these films was as follows:

Temperature Range 74°F to 84°F
 Relative humidity 45 - 65%
 Exterior hermetic packaging was opened
 Films were stored flat due to space considerations

A tabular listing of the out of date films tested follows.

<u>Film Type</u>	<u>Packaging</u>	<u>Expiration Date</u>	<u>Original Base + Fog</u>	<u>Current Base + Fog</u>	<u>Sensitivity %</u>	
					<u>Old</u>	<u>New</u>
Kodak M	Bulk	June 1980	0.10	0.13	1.7	1.8
Kodak M	Redipak	July 1980	0.10	0.13	1.9	1.9
Kodak T	Bulk	Jan 1981	0.11	0.18	1.6	N/A
Dupont 55	Bulk	July 1981	0.14	0.20	2.2	2.0
Dupont 55	Redipak	Nov 1981	0.14	1.0	3.3	1.9
Dupont 65	Bulk	July 1981	0.21	0.22	2.0	N/A
Dupont 65	Redipak	July 1981	0.21	0.22	2.0	N/A

5. Air Force Films

Films were received from four Air Force units in various locations throughout the world. A listing of these locations, types of film, and stated storage conditions are:

<u>Location</u>	<u>Kodak Film Type</u>	<u>Storage Conditions</u>
Kunsan AFB, Korea	AA daypack	Standard Lab
Ft. Wayne, Indiana	M & AA daypack	Standard Lab
Patrick AFB, Florida	M & AA daypack	Standard Lab
Kadena AFB, Japan	M & AA daypack	Refrigerated

* Evaluation and Comparison of Industrial Radiographic Film Characteristics used for NDI. Williams, R.D. and Teller, C.M., 1980; SwRI Project No. 15-5607-803, Contract No. DLA 900-79-C-1266 (CLIN 001AB).

The above films were tested using the same techniques used throughout this program. A listing of results follows. Films are identified by unit number and exposure number.

<u>Film No.</u>	<u>Type</u>	<u>Base + Fog</u>	<u>Date Recv'd</u>	<u>Sensitivity @ D ≈ 2.0</u> %
122TFW-1	M Day Pack	0.20	4/75	1.6
122TFW-2	AA Day Pack	0.31	4/80	1.9
122TFW-3	AA Day Pack	0.315	9/79	2.0
122TFW-4	M Day Pack	0.17	9/76	1.6
549-1	M Day Pack	0.15	9/82	1.7
549-2	AA Day Pack	0.21	5/81	2.0
8CRS-1	AA Day Pack	0.22	3/82	2.0

Test exposures were made on all the above Air Force films on 3 March 1983. The films from Kadena AFB arrived later in the program and were not tested due to a subsequent failure of the x-ray tube head.

V. CHEMICAL TESTING

As requested in the Statement of Work, an investigation of possible chemical tests applicable to field determination of film aging was carried out. An extensive computer search of Chemical Abstracts was carried out with negative results. Additionally, searches of available manufacturers literature and personal communications with individuals in the fields of Chemistry, Photography and Radiography indicate that the only known method is the actual processing (developing) of an unexposed candidate film for determination of base plus fog level. Increases in base plus fog level are detrimental in that they decrease the dynamic range of a particular film type. However, except for gross increases, base plus fog level should not be used as the sole criteria for accepting or rejecting radiographic films because of the widely varying dynamic ranges exhibited by different film types.

As further requested by sponsor representatives at the first in progress review, a determination of actual silver content for the various films was desired. Accordingly, Dr. Leon Adams of the Division of Chemistry and Chemical Engineering at SwRI was contacted and he designed a test to determine this quantity. For the tests, sample coupons of each of the films were prepared having exactly 10 cm^2 surface area (i.e. 5 cm^2 each side for double coated films). Mr. Bill McMahon of the Division of Chemistry and Chemical Engineering conducted the actual testing. The following is a description of the method and a tabulation of results.

Determination of Silver Content of X-Ray Film

1. Place a film sample of known area (10 cm^2 both sides) in a 100 ml beaker, add 10 ml of 1:1 nitric acid and warm on a steam bath or hot plate until the emulsion begins to loosen and sluff off of the film.
2. Using stainless steel tweezers, for handling the film, rub the surface of the film with a rubber policeman until all of the emulsion has been removed.
3. Using the tweezers remove the film from the beaker and rinse the film, tweezers and the rubber policeman with a small quantity of demineralized water, collecting the rinses in the sample beaker.
4. Add 5 ml of concentrated sulfuric acid, a silica boiling stone, partially cover the beaker with a watch glass and boil on a hot plate until the liquid volume has been reduced to 6-8 ml.
5. Add 5 ml concentrated nitric acid, cover with a watch glass and boil on the hot plate until all of the silver halide has been dissolved. Cool to room temperature.
6. Dilute sample to approximately 75 ml with water and titrate potentiometrically with standard 0.014 N Sodium Chloride solution, using a Sargent-Welch recording Titrator, Model DG, equipped with silver-silver chloride and calomel electrodes, until the end has been passed by at least 3 ml of titrant (The titrimeter plots millivolts vs ml of titrant on the chart paper.)

7. With the aid of a straight edge determine the exact end point, in the break of the curve, using standard techniques for interpretation of potentiometric titrations.

8. Calculation

$$\text{mg Silver/cm}^2 = \frac{\text{ml NaCl} \times 0.0141 \times 107.9}{10}$$

where 107.9 is the molecular weight of Silver

$$= \text{ml NaCl} \times 0.1521$$

Tabulation of Results

<u>No.</u>	<u>Sample Description</u>	<u>ml of 0.0141NNaCl</u>	<u>mg Ag/cm²</u>
1	Dupont Type 75	4.94	0.75
		4.95	0.75
2	Dupont Type 55	4.90	0.75
		4.90	0.75
3	Dupont Type 65	5.08	0.77
		5.47	0.83
		5.33	0.81
4	Kodak Type AA	6.31	0.96
		6.34	0.96
5	Kodak Type M	6.16	0.94
		6.09	0.93
6	Kodak Type T	6.10	0.93
		6.16	0.94
7	Gevaert D7	6.12	0.93
		6.21	0.94

VI. FILM NOISE

A. Grain Size Determinations

Attempts at direct emulsion grain size measurements were made using the scanning electron microscope (SEM) facilities of the Division of Mechanical Sciences and Engineering at SwRI.

Initial SEM photographs made on uncoated film surfaces were greatly distorted due to the de-focusing effect of electron charge on the non-conducting emulsion surface. Additional photographs were attempted after coating the emulsion surface with a thin conducting layer to carry off this electronic charge. Results of these tests indicated that when this conductive coating was sufficient to suppress charging, the masking effect of this coating would preclude attempts at grain size measurements.

Consideration of additional, more sophisticated, approaches was given but abandoned due to the fact that one of the primary purposes of this study was to develop a simple, field applicable, test for film quality.

B. Electronic Film Scanning

Graininess is defined as the visual impression of non-uniformity of density in a radiographic (or photographic) image. Normally in radiography with fast films exposed to high kilovoltage radiation, the graininess is apparent to unaided vision; with slow films exposed to low kilovoltage radiation, moderate magnification is necessary to make it visible. Literature has also indicated that graininess also increases with film degradation. To test for the graininess in the film tested in this program, a modified version of a previously used system was employed. The modifications to the system were, to magnify the video image to the maximum extent possible within the limitations of the system (to show the grain as a sinusoidal ripple on the trace) and to utilize a Tektronix 7854 digital oscilloscope for evaluation purposes. The Tektronix 7854 oscilloscope was selected because of its capability to digitize and store an integrated waveform. Integration eliminates or minimizes electronic noise. For this study, ten (10) waveform integrations were used.

Figure 5 is the block diagram of the system comprising six major components:

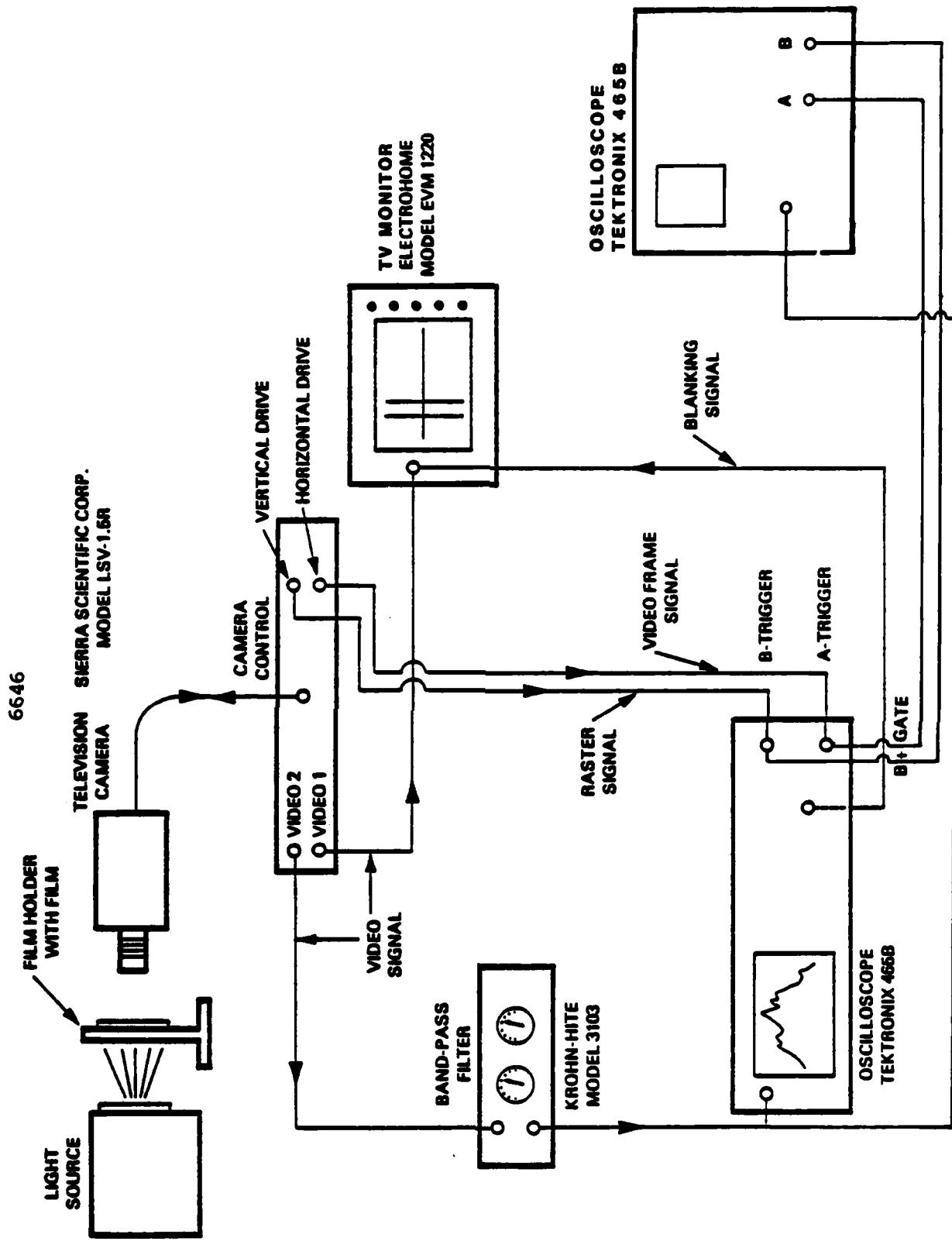
- Film viewer
- Television camera
- Camera control unit
- Television monitor
- Oscilloscope
- Filter

System operation is based on a "line-grabber" concept. Each raster line on the TV monitor is produced by sweeping an electron beam across the screen. A varying voltage controls the electron image and, thereby, the local brightness of the line. It is possible to extract the voltage signal to one of the lines (the "line-grabber" concept) and use that signal for other purposes. In this case, the signal is fed to the oscilloscope where it is displayed on the cathode-ray tube (CRT) where the voltage deflection of the trace is a direct analog of film density. On the monitor, a white line replaces the normal raster.

As shown in Figure 6, an X-ray film is placed in the film holder and illuminated by the light source. The electronic X-ray image (see Figure 7) is picked up by the camera and fed to the monitor and oscilloscope through the camera control. Trigger signals are sent to the oscilloscope from the control to synchronize the oscilloscope and television systems. After the operator selects the image of interest on the monitor, he changes the "Delayed Sweep" until the selected raster line intersects the image. A blanking signal from the "B-Gate" on the oscilloscope causes the selected raster line to be white. At the same time, the voltage signal for that raster is displayed as a trace on the oscilloscope screen where the trace deflections are measured. Deflections of the oscilloscope trace are proportional to the varying raster voltage signal and are, therefore, proportional to the image brightness. At the high gain settings used, the "graininess" is displayed as a ripple voltage with the periodicity, (frequency) being representative of the graininess of the film.

The frequency of the "grain" signal is then displayed on the Tektronix 7854 scope and indicates the relative graininess of the film i.e., the lower the frequency, the larger the "grain" of the film and vice versa. Figures 7a through 7c are examples of this type of evaluation. As can be seen by the photographs, the slower films did have a higher frequency than the faster films, thus a smaller "grain". Since the composite signal displayed on the oscilloscope also contains "frequency" component contributions from film defects other than grain size alone, such as clumping, the total effect should be more properly stated as noise. High background noise in radiography may be considered in an analogous sense to electronic noise wherein an information signal (defect image) may be totally obscured by high noise levels.

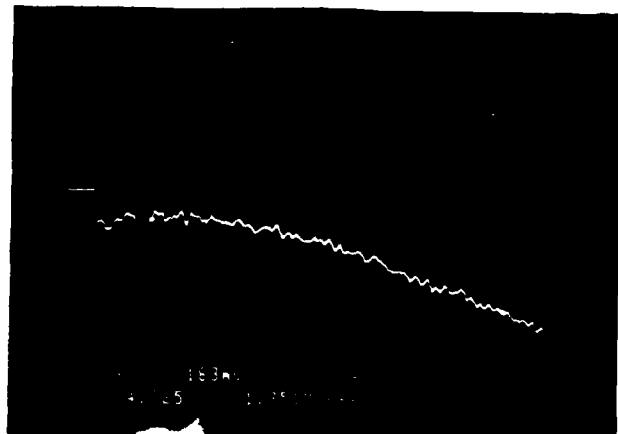
While the above provides an effective demonstration of concept, considerable additional study would be required to demonstrate feasibility in regard to determination of film quality. The additional work required is considered to be beyond the time and budgetary constraints of this current project.



PICTURE 6. BLOCK DIAGRAM OF RADIOPHASIC ANOMALY MEASUREMENT SYSTEM

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A.



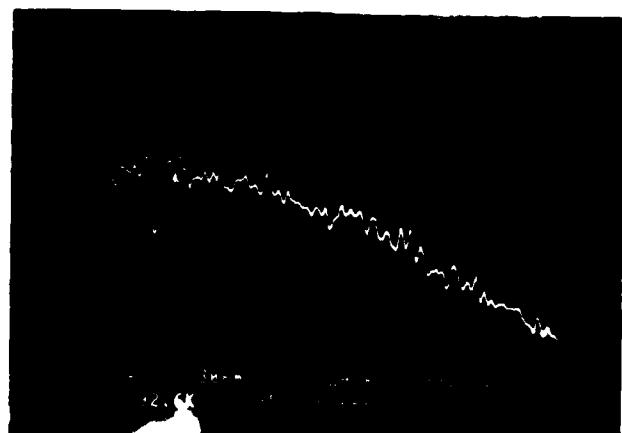
KODAK TYPE M

B.



KODAK TYPE AA

C.



DUPONT NDE 75

X-RAY ELECTRONIC NOISE

FIGURE 7. X-RAY NOISE TRACES

VII. DISCUSSION OF RESULTS

A. Environmental Testing

The results of these tests indicate that a warm, high humidity environment is extremely detrimental to film stored in opened boxes where the environmental seal has been broken. Films stored in unopened boxes where the environmental seal has not been violated would, in all probability, react in a manner similar to that of films stored in the high temperature (Desert) environment. That is, a thermally induced increase in base plus fog densities would occur with time. Elevated temperature, dry film storage, over extended times, will cause increases in base plus fog levels and eventual loss in film sensitivity through reduction in the original dynamic range. Short term storage or use of films in the presence of high temperatures apparently will not cause measurable changes in film characteristics. X-ray films exposed to sub-zero temperatures showed no changes in original characteristics during the time span of this study. Long-term storage at these temperatures should probably be avoided due to possible dehydration of the film emulsion. The new films stored under ambient lab conditions showed no indication of a change in characteristics. However, the out of date films tested, as shown in IV, B, 4. above, did show increases in base plus fog level with one, Dupont NDT 55, Day-pack, showing a base plus fog of 1.0 H&D unit and decreased sensitivity. The other old films in this group demonstrated sensitivities comparable to films in the new group and would be entirely suitable for use in radiographic inspections. Densities, as measured at equal step thickness, also indicate no change in film speed, when compared to new films used in this project.

B. Chemical Determinations

From the results shown in the quantitative silver determinations of Section V above, it is obvious that the quantity of silver is not the primary determinant of film speed for X-ray films. It is of interest to note the relatively large difference in silver content of films produced by Kodak as compared to Dupont films, these films having similar published speed and grain size characteristics.

C. Film Noise

As previously discussed in Section VI above, the use of the scanning electron microscope (SEM) did not prove to be a viable approach for direct grain size measurement. Other more sophisticated approaches to direct grain size determination were not considered since one of the main objectives of this study was to explore simple, field applicable, tests for X-ray film integrity.

The second approach, detailed in Section VI, does show promise. The digitizing of the data from a single scan and resultant display of frequency content is directly related to film noise, in that, the higher the frequency content, the lower the noise. This noise includes grain size, clumping and other anomalies directly related to film quality. Further study of this approach, although not possible under this current program, should be carried out.

VIII. CONCLUSIONS AND RECOMMENDATIONS

Studying the results of the series of tests performed during the period of this investigation the following conclusions may be deduced.

A. Storage Environment

1. High Humidity at Elevated Temperature (Tropical)

This is a storage environment which definitely is detrimental to films stored in opened basic packaging where the environmental seal, provided by most manufacturers, has been broken. It cannot be stated, as a result of these tests, that film stored in the original unopened boxes and having the environmental seal intact, would degrade in the manner observed during these tests. Degradation of these films in unopened boxes, would most probably occur in a manner similar to that of high temperature storage as explained below.

2. High Temperature, Low Humidity (Desert)

Long-term storage of films in an elevated temperature environment should be avoided although relatively short term exposure to higher temperatures, such as may be incurred during shipping or field use, would probably not produce a measurable film degradation. Thermally induced fog is a non-reversible cumulative effect which is analogous, in many respects, to the actual exposure of a film, in that the higher the source intensity, the shorter the exposure for a given density. Thermal fogging of all films under simulated desert conditions was evident after four months of exposure. Longer exposures would continue to increase this fog and decrease film sensitivity through reduction in the dynamic range of the film.

3. Sub-Freezing Storage (Arctic)

No apparent damage to film in opened packaging was detectable over the course of this study. However, longer periods of storage could lead to excessive drying of film emulsions stored in opened packages. Films stored in unopened packages, with environmental seals intact, would probably not be damaged and most likely would demonstrate greatly extended shelf life due to the reduced rate of chemical reaction which occurs at low temperatures.

4. Ambient Storage

New films in opened packages, stored under standard laboratory conditions for the period of this program, showed no evidence of deterioration in film characteristics. Old films, considerably past manufacturers expiration dates, stored in essentially identical conditions for four years at SwRI, did show some changes in characteristics. The observed change in these old films was an increase in base plus fog levels. With the exception of one film in this group, Dupont NDE 55, Daypack, testing showed an acceptable level of sensitivity for critical inspections. For the Dupont film, base plus fog had increased to a level of 1.0 H&D unit and sensitivity had decreased to 3.3% along with greatly reduced dynamic range (Dynamic range being here specified as the density difference between base plus fog and the greatest useable density obtained upon exposure. Other old films, received from Air Force units,

also showed increases in base plus fog levels, however, these same tests also demonstrated acceptable sensitivities for all Air Force films. These results indicate that laboratory storage conditions at the responding Air Force bases were at least adequate for extended film storage. (One of these films had been stored since April 1975!)

B. Recommendations for Extended Storage

1. Warehouse Storage

Extended film storage in warehouses where temperatures exceed 85°F is not recommended. Wherever possible, storage temperatures below 75°F should be maintained and where extremely long storage periods are anticipated, refrigerated storage, 35° to 45°F, is recommended for extended film life.

2. In Use Storage

In laboratories where a high use factor is experienced, storage at average ambient conditions of 72° -80°F and relative humidity less than 50% is entirely satisfactory. For laboratories where relative humidity exceeds 75% with temperatures over 85°F for a majority of the time, refrigerated storage should be given serious consideration to avoid premature film failure.

For laboratories with a low use factor refrigerated storage is definitely recommended to provide extended film life.

C. Recommendations for Shipping

1. Surface Shipment

For surface shipment of radiographic films it is recommended that refrigeration be required where estimated route temperatures may exceed 95°F. If this is not possible, a temperature monitor should accompany the shipment to provide a rudimentary time versus temperature history to indicate whether testing is required before using this film in critical inspections. It is believed these precautions are necessary because temperatures at times may exceed 120°F inside of both ship holds and over the road vans.

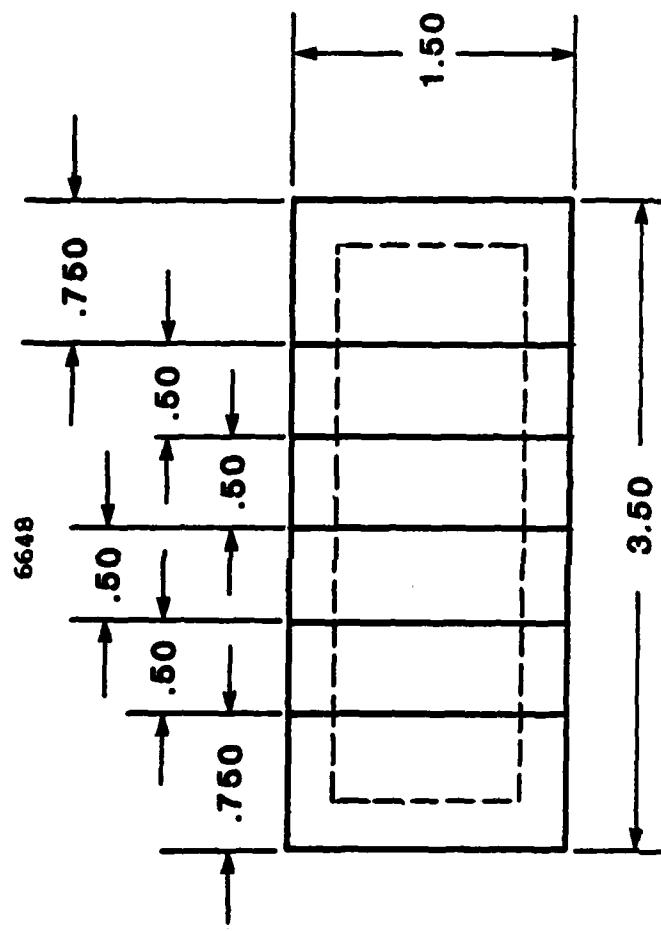
2. Air Shipment

Due to the relatively short time periods involved in air shipment it is not believed necessary to provide for any special handling other than normally required for photo-sensitive materials.

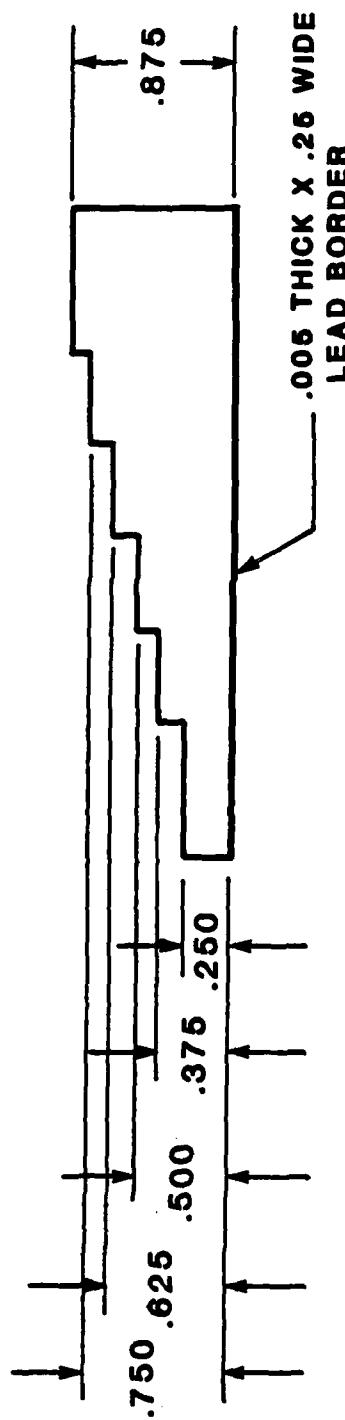
D. Recommendations for Film Testing

1. Step Wedge Test for Sensitivity

For film sensitivity determinations comparable to those accomplished during this study, exposures should be made at 110 kilovolts in order to provide similar radiation quality incident on the step tablet. Using the step tablet of Figure 8, expose the test film so that a resultant density of 2 H&D units will occur between the 0.500 inch and the 0.625 inch steps of the step tablet after processing. An additional, unexposed, sheet of the film undergoing test should be processed simultaneously with the exposed film.



NOTE:
ALL STEP TOLERANCES $\pm .001$



STEP WEDGE FOR SENSITIVITY DETERMINATIONS

2024 Aluminum Alloy

FIGURE 8. STEP WEDGE FOR SENSITIVITY DETERMINATIONS

After processing, use a calibrated densitometer to make and record at least three densitometer readings for each thickness step of the step wedge. Average the three readings for each of the steps and prepare a tabulation of density vs. step thickness as follows:

Density	Thickness in Inches
3.75	0.250
2.83	0.375
2.22	0.500
1.79	0.625
1.45	0.750
1.20	0.875

Note: Densities shown in the above tabulation are taken from Figure 5 of this report and are shown for illustration.

Now take, record and average at least four readings from the unexposed processed film sheet. This is average base plus fog for the film undergoing test.

At this point, refer to the instructions in Section III of this report and either calculate the film sensitivity or plot the results on the recommended graph paper and use the overlay (Figure 4) to determine approximate film sensitivity. If only calculation is used it would be advised to calculate film sensitivities at each pair of steps on either side of the first pair. This will provide a check for data consistency.

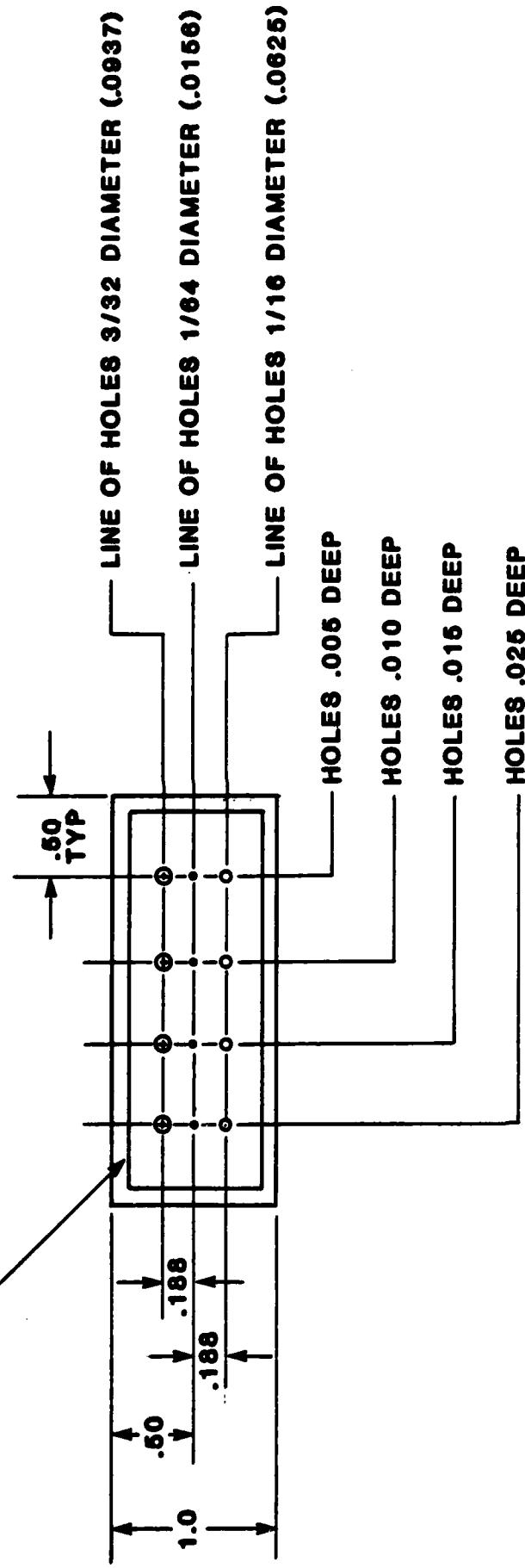
2. Special Sensitivity Penetrometer Test

A direct reading sensitivity test penetrometer has been designed. A line drawing of this penetrometer is shown in Figure 9. The families of flat bottom holes represent exact thickness percentages of the test block. Hole depth vs. percent sensitivity is listed below:

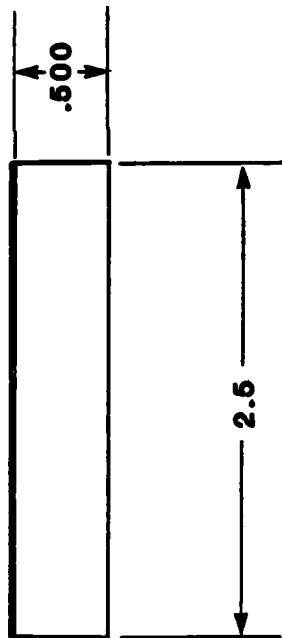
<u>Depth</u>	<u>Percent Sensitivity</u>
0.005 inch	1.0
0.010 inch	2.0
0.015 inch	3.0
0.025 inch	5.0

For use a test film is exposed to a desired density of 2.0 to 3.0 H&D units at a kilovoltage appropriate to the expected inspection requirements. After processing, the film may be observed using a high intensity viewer and optical magnification if required. The approximate film sensitivity may then be determined directly.

6649

LEAD BORDER 3/16 WIDE X .006 THICK

NOTE:
ALL HOLES TO BE FLAT BOTTOM
AND WITHIN 5% OF STATED DEPTH.
ALL DIMENSIONS ARE IN INCHES



SENSITIVITY TEST PENETRAMETER

2024 Aluminum Alloy

FIGURE 9. SENSITIVITY TEST PENETRAMETER

An added advantage of this type of penetrometer is that a qualitative measure of film resolution may also be made. That is, are all hole sizes readily apparent at the minimum sensitivity observed?

3. Resolution Capability of Radiographic Film

Until this point little has been said about the resolution capability of films. Different films, with equal sensitivity, will not achieve the same capabilities for resolving the small defects frequently searched for in critical inspections. This is due to the amount of film noise. Film noise is caused by grain size, "clumping" and other sources. Manufacturer's descriptions of radiographic films generally include terms such as, extra fine grain, fine grain, medium fine grain and relatively fine grain. Films with the smaller grain size demonstrate good resolution capability however, increases in base plus fog level, "clumping" and other mechanisms will tend to decrease this resolution capability.

Simple qualitative testing for resolution may be accomplished using a special penetrometer as described in VIII, D, 2. above. A more definitive approach would be to use a standard photographic resolution test pattern as shown in Figure 10, and make contact prints, using a collimated light source for exposure. Examination of the resultant "print" will provide direct information as to film resolution capability. For optimum results, both a negative and a positive transparency of the test pattern should be used because of the thickness of radiographic emulsions. Also a black non-reflecting backing should be used during exposure.

E. Recommendations for Film Life Extension

1. Life Extension Through Testing

Since, as has been demonstrated by this study, simple, field testing of radiographic films can be accomplished with reasonable accuracy. It is recommended that no film, regardless of age, be discarded unless tests are accomplished to determine level of film degradation. Establishment of precise levels of film degradation for discontinuation of use is not possible. However, the following guidelines, based on several types of inspection should be helpful.

Critical Inspections - Fine Cracks

Only extra fine grain or fine grain film with a demonstrated sensitivity of better than 2% at a density of 2.0 H&D units and having base plus fog levels no greater than a factor of 2 above that of new film of the same type should be used for this type inspection. Films must also demonstrate resolving power capable of detecting defects in the size range expected.

Critical Inspection - De-lamination & Gross Cracking

For this type of inspection fine to medium fine grain films with a sensitivity of 2% or better at a density of 2.0 H&D units and base plus fog level should not exceed a value of 0.4 H&D units. Film resolution should be adequate to detect gross cracking.

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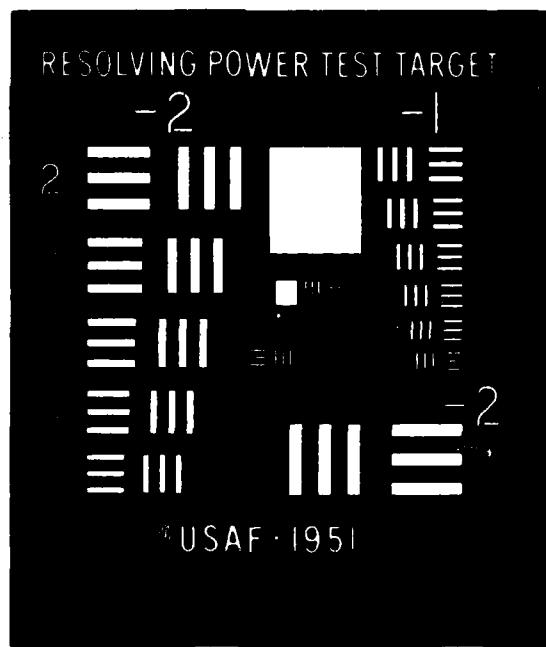
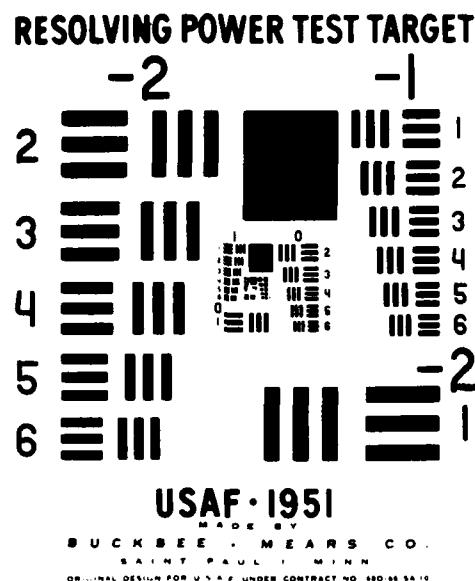


FIGURE 10. RESOLVING POWER TEST TARGET

Inspection - Survey

For general survey inspections such as parts placement, loose nuts and bolts, misplaced tools etc., a film sensitivity better than 3% and a base plus fog level no greater than 0.7 H&D units is recommended.

Inspection - Training

For training purposes, films in all categories above should be utilized to provide job/inspection correlation. However, for purposes of film classification, training films should have a sensitivity better than 5% and a base plus fog level not to exceed 1.0 H&D unit.

Above these levels all film should be salvaged.

2. Life Extension by Improved Storage

In commercial practice, film life extension has long been accomplished through use of refrigerated storage. A usage factor is usually applied in arriving at a decision to use refrigerated or ambient (laboratory) storage environments.

It is recommended that for laboratory units with a low use factor, refrigerated storage facilities for opened film packages be used. This would be particularly useful where high ambient temperatures and humidity are experienced. For units with a high use factor ambient storage is in most cases entirely adequate.

FILME

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DTIC